Attachment 2

Laboratory PE Sample Results

Procedure:

1/24/01 Two PE Samples were purchased from Environmental Resource Associates [ERA]--one for total mercury, and one for TCLP mercury.

1/28/01 PE Samples were delivered to Veritech, and PE Certified Values were delivered to ME DEP.

Veritech analyzed PE samples according to SOPs provided in the ME Lamp Study QAPP.

Analysis:

Analysis for TCLP mercury was conducted 2/5/01. PE sample is Environmental Resource Associates TCLP Metals in Soil, Lot No. 85009. Veritech results were faxed to ME DEP 2/16/01 and are tabled below:

Veritech Value	Certified Value	% Recovery	Acceptance Range
1.8 ppm	2.04 ppm	88.2%	0.973-3.10 ppm

Analysis for total mercury was conducted 2/7/01 and faxed to ME DEP 2/16/01. PE sample is Environmental Resource Associates Priority Pollution Inorganic Soils lot no. 248. Robin Jetter [Veritech Quality Assurance Officer] called me to say she found a calculation error and would fax a corrected copy of results. A corrected copy was received by ME DEP 3/2/01, and is tabled below:

Veritech Value	Total Concentration**	% Recovery	Acceptance Range
19.7 ppm	6.47 ppm	304%	4.19-8.23 ppm

**Total concentration was used to document % Recovery because certified value was for EPA methods 3051, and the extraction method used in this analysis was a modification based on whole lamp analysis.

Result for a 0.6 g sample [per phone conversation 4/5/2001] analyzed by traditional SW 846 method 7471A yielded a result of 8.02 mg/Kg.

Results:

Results for the TCLP mercury test show that this method is under control, and the laboratory is proficient for this analysis.

Results for total mercury are outside acceptance ranges. ME DEP cannot accept that Veritech is proficient for this analysis based on these results. Note that a 0.6 g sample analyzed by the traditional method yielded results within the acceptance limits demonstrating lab proficiency with the traditional method.

Deb Stahler called Robin Jetter to discuss results of these analyses. Discussions ensued with several technicians, chemists, and the quality assurance officer to evaluate the cause for high total mercury results.

Follow Up:

Two theories emerged. One is that the soil PE provided by ERA is certified for listed methods only, and that this sample may not be an appropriate PE for whole lamp analysis of mercury. The second is that interference from free chlorine may be greater in whole lamp analysis due to the elevated volume of digestion acid [HCl + HNO3] used in sample preparation.

To test these two theories ME DEP purchased an aqueous spiking solution from ERA. The spiking solution was shipped to Veritech 5/9/01, and the certified value of the spiking solution was sent to ME DEP. ERA lot number 0507-01-05 [NIST SRM 3133] certified value is 4.00 mg/ml. Veritech performed the following tests:

- Spike a reagent blank with 1 ml of the spiking solution and perform the analysis according to SOP 4219 as provided in the ME Lamp Study QAPP.
- Spike a reagent blank with 1 ml of the spiking solution and perform the analysis according to SOP 4219 as provided in the ME Lamp Study QAPP with one modification. Modification is to bubble inert gas through the digested sample prior to sample reduction and analysis.

Results of this testing is tabled below:

Sample	**Result**	**True Value**	**% Recovery**	**Acceptance Range**
LFB no purge	12.0 mg	4.0 mg	300	75-125% TV
LFB purge	12.0 mg	4.0 mg	300	75-125% TV

Results from this round of PE testing suggest that a mathematical error exists somewhere in the process. Both this and the last PE samples had recoveries in the range of 300% Robin Jetter and Dave Mathews reviewed the process for mathematical errors, and concluded that dilution error probably accounted for much of this error. The working range of the test is 10-1000 ppb. Lamp results are expected to be in the ppm range and PE samples were selected to give results in the mg range. This range necessitates large dilutions, which can multiply errors by the dilution factor.

On 6/8/01 Deb Stahler traveled to Veritech Laboratory to review the total mercury test. Dave Mathews, Robin Jetter, Stan Gilewicz, and Deb Stahler met to discuss problems with the PE sample results. During the meeting Dave Mathews revealed that the calibration criteria was often hard to achieve, and he believed this was due to the large calibration range. Responses for mercury concentrations over 500 ppb appeared to cause the actual curve to deviate from a linear response. Stan Gilewicz proposed, and all concurred that Dave should try a second order calibration curve to solve the problem. If the actual calibration results do not closely follow the second order curve, the calibration range should be truncated to 500 ppb. Dave Mathews proposed, and all concurred that the initial extract volume would be changed from 250 ml to 100 ml for ease of calculations. Dilutions are expected to be in the range of 1/1000.

Stan Gilewicz escorted Deb Stahler on a tour of the laboratory. The laboratory appeared neat and well equipped. Veritech is primarily a remediation laboratory analyzing mostly soil and sediment samples. In addition, Philips Lighting has lamp samples analyzed for

TCLP mercury at Veritech, so it is equipped with 6L and 8L TCLP extraction apparatus. Equipment for preparation and analysis of mercury samples appeared to be in good working order. There is no back up instrument for mercury analysis, but the laboratory does maintain a service contract for the analyzer.
Lamps in the USPS shipment boxes received from MEDEP were stored in a warehouse room that appeared to be clean. Boxes were in good condition, and lamp breakage from shipment appeared minimal.

Follow up:

Several calibration ranges were evaluated, and it was found that the calibration upper limit was in the range of 200 ppb. Instrument calibration was changed in the SOP. The new calibration scheme is 5, 10, 25, 50, 100, and 200 ppb. Using the PE sample [4 mg/ml] as an SRM, Dave Mathews prepared and analyzed samples following the modified protocol. Results are as follows:

Sample	**Result**	**True Value**	**% Recovery**	**Acceptance Range**
ERA 0507-01-05	4.6 mg	4.0 mg	115	75-125% TV

An additional custom spiking solution was purchased from ERA [lot no. 0612-01-06] to be used as a single blind PE sample. On 7/18/01 Veritech prepared and analyzed the PE according to the modified protocol. Results are as follows:

Sample	**Result**	**True Value**	**% Recovery**	**Acceptance Range**
ERA 0612-01-06	2.27 mg	3.0 mg	75.7	75-125% TV

On 7/19/01 ME DEP confirmed that the total mercury PE sample was within acceptance limits and Veritech was authorized to analyze the first sample delivery group samples.